Patent Publication No. 7-22692 (B2)

(24)Date of publication of patent : 15.03.1995
(21)Application number : 63-194338
(22)Date of filing : 05.08.1988
(65)Published Patent Application No. :2-43939
(43)Date of Publication of Patent Application :14.02.1990
(51)Int.CI. : B01J 3/04
C30B 7/10 C30B 29/18

Title of the Invention : CONTAINER FOR HYDROTHERMAL SYNTHESIS
Inventor(s) : Kotaro CHIBA et al.
Applicant : Japan Steel Works, LTD.

[Claim(s)]

[Claim 1] it has micropore also on the buffer room which prepares a tubed gap, is built in in the body of a container heated from the outside, an inside prepares micropore in the lid of the container liner container formed with noble metals, such as silver, gold, and platinum, prepares at least the wall which covers this micropore in a top-face [of a lid], or inferior-surface-of-tongue side, and is formed with a lid and a wall, nothing, and this wall surface -- carrying out -- **** -- the container for hydrothermal synthesis characterized by things.

[Claim 2] From the outside, in the body of a container heated, a tubed gap is prepared and it is built. An inside at least Silver, Nothing [which prepare micropore in the lid of the container liner container formed with noble metals, such as gold and platinum, prepare the wall which covers this micropore in a top-face / of a lid /, or inferior-surface-of-tongue side, and are formed with a lid and a wall / the buffer room and nothing], have micropore also on this wall surface -- carrying out -- in the container for **** hydrothermal synthesis -- the space volume of a buffer room -- The container for hydrothermal synthesis characterized by setting up more thinly than the

alkali concentration in a container liner container the concentration of the alkali solution with which filled up the real space volume of said tubed gap, and the real space volume of a container liner container with the alkali solution as a respectively fixed volume rate, and they were filled up in the buffer room and the tubed gap.

[Claim 3] The 1st term of a patent claim which made the tubed gap between the body of a container, and a container liner container fill up with the permeability matter with good thermal conductivity, or the container for hydrothermal synthesis given in the 2nd term.

[Detailed Description of the Invention]

(Field of the Invention)

This invention relates to the container for hydrothermal synthesis used for manufacture of synthetic rock crystal etc.

(Prior art)

An example of the conventional container for hydrothermal synthesis is shown in Fig. 4 . For the body of a container, and 22, as for a clamp and 24, a lid and 23 are [21 / a convection-current control strip and 25] heaters, and 26 is a thermocouple each. Seed A and a raw material B are held in the body 21 of a container, and it is immersed in the strong-base solution heated at the heater 25. If the case of synthetic rock crystal is taken for an example, since it is used by the temperature of 350-400 degrees C, and the pressure of 1000-1500kg/cm2, the metallic material the body 21 of a container excelled [metallic material] in high intensity, high toughness, and corrosion resistance will be used.

Although the product manufactured by hydrothermal synthesis has many applications to fields, such as an electron and optics, and they will be in a miniaturization and the inclination by which thin fasciation is carried out increasingly from now on, it has mixing of a foreign matter, using it as the key factor which makes a product crude. especially, therefore, body of container 21 inside erodes in a strong-base solution -- having -- bitter taste -- a dynamite -- an iron compound is produced, it is the greatest trouble that Fe+ ion mixes to a product, and various measures are taken.

Although there is a method of isolating the front face of Seed A as an example, it is not enough, and growth is checked and, as for the protected field, productive efficiency falls. Moreover, the container liner container which covered the inside of the body 21 of a container with silver, gold, platinum, etc., or was manufactured with the above-mentioned noble metals is made to build in the body 21 of a container, and preventing the generating of Fe+ ion itself is also made.

(Object of the Invention)

When covering the inside of the body 21 of a container with silver, gold, and a platinum cylinder From from [when securing adhesion with the wall of the body 21 of a

container], means, such as water pressure expansion or explosive bonding, are required. Therefore, manufacture in the structure of the body 21 of a container instead of very difficult **** **, In making the container liner container which produced the problem in maintenance of the sheet surface of a seal part, and was manufactured with ****, gold, platinum, etc. build in the body 21 of a container It is necessary to make the internal and external pressure of a container liner container equate, and, for that, it is important to hold identically the internal and external volume rate of a container liner container so that one-sided external pressure may not act on a container liner container. The deer was carried out and the applicability of above-mentioned both was limited to the small container for both experiments.

(The means for solving a technical problem)

This invention is made in view of the above-mentioned situation, the container for hydrothermal synthesis which made it large-sized and fitted industrial use is offered, and the configuration is as follows. that is, the 1st invention has micropore also on the buffer room which prepares a tubed gap, is built in in the body of a container heated from the outside, an inside prepares micropore in the lid of the container liner container formed with noble metals, such as silver, gold, and platinum, prepares at least the wall which covers this micropore in a top-face [of a lid], or inferior-surface-of-tongue side, and is formed with a lid and a wall, nothing, and this wall surface -- it carries out and it is a container for **** hydrothermal synthesis.

Moreover, in the body of a container heated from the outside, the 2nd invention prepares a tubed gap and is built in. Micropore is prepared in the lid of the container liner container in which the inside was formed with noble metals, such as silver, gold, and platinum, at least. Nothing [which prepare the wall which covers this micropore in a top-face / of a lid /, or inferior-surface-of-tongue side, and are formed with a lid and a wall / the buffer room and nothing], have micropore also on this wall surface -- carrying out -- in the container for **** hydrothermal synthesis -- the space volume of a buffer room -- It is the container for hydrothermal synthesis which set up more thinly than the alkali concentration in a container liner container the concentration of the alkali solution with which filled up the real space volume of said tubed gap, and the real space volume of a container liner container with the alkali solution as a respectively fixed volume rate, and they were filled up in the buffer room and the tubed gap.

And the tubed gap between the body of a container and a container liner container may be made to fill up with the permeability matter with good thermal conductivity in both the above-mentioned invention.

(Work for)

Next, an operation is explained.

A seed and a raw material are paid into the container liner container of the container for hydrothermal synthesis, the body of a container is heated from the outside, and hydrothermal synthesis is performed.

Since the inside is formed with noble metals at least in that case, Fe+ ion is generated within the body of a container, and a container liner container does not affect a seed.

Moreover, since the tubed gap of the body of a container and the inside of a container liner container are open for free passage by the micropore prepared in the lid of a container liner container, and the wall surface of a buffer room, the pressure of container liner container inside and outside is balanced, and a container liner container does not receive one-sided external pressure. Therefore, a container liner container is not distorted or it does not damage.

Moreover, therefore, the pressure with the inside of a tubed gap and a container liner container is balanced substantially to use the concentration of the alkali solution with which was filled up with the alkali solution made into a respectively fixed volume rate, and the buffer room and the tubed gap were filled up for the space volume of the buffer room of the aforementioned container for hydrothermal synthesis, the real space volume of a tubed gap, and the real space volume of a container liner container, making it thinner than the alkali solution concentration in a container liner container. And if pressure variation arises, the pressure in a tubed gap and a container liner container is balanced through both micropores.

Although the ARUKARU solution of the minute amount containing Fe+ ion trespasses upon the buffer interior of a room from micropore when the pressure in a tubed gap rises Since it mixes with the alkali solution of the buffer interior of a room, Fe+ ion is diluted and it invades into a container liner container The effect which it has on a seed is minute, in addition since the concentration of the alkali solution with which the tubed gap was filled up is thin, the inside of the body of a container is hard to be eroded, and the effect on a seed is conjointly mitigated further with the yield of Fe+ ion decreasing.

Furthermore, if the tubed gap is filled up with the permeability matter with good thermal conductivity, heat transfer from the body of a container to a container liner container will be made good, and the pressure in a tubed gap will be kept equal.

(Example)

The example of the container for hydrothermal synthesis concerning this invention is explained based on a drawing.

In Fig. 1 , 1 is a body of a container heated from the outside, and consists of a metallic material excellent in high intensity, high toughness, and corrosion resistance. 2 is the lid of the body of a container, it infixes packing 2a and the body 1 of a container is equipped with it removable by two or more clamps 3.

the ingredient which 4 is a cylinder-like-object-with-base-like container liner container and made noble metals the cladding metal at insides, such as products made from noble metals, such as silver, gold, and platinum, or Ti (titanium) material, -- intermediary **** [from]. lid 4a and bottom plate 4b of a removable container liner container -- from the same ingredient -- becoming -- this lid 4a -- mostly, micropore 4a' is provided in the center section so that it may **** to Fig. 2 . In the condition that a pressure does not act, this micropore 4a' is magnitude down which a liquid therefore does not flow by self-weight to surface tension, and is specifically 0.4mmphi extent. And intermediary-ed cube type member 5a is fixed for micropore 4a' on the top face of lid 4a of this container, the buffer room 5 is formed in it, and micropore 5b of 0.4mmphi extent is prepared also in the top plate of this core box member 5a. In addition, let the location of this micropore 5b be a location higher than the oil level of the liquid with which the buffer room 5 is filled up.

Moreover, even if there are few buffer rooms 5, the inside is formed with noble metals.

The container liner container 4 which consists of the above-mentioned configuration prepares tubed gap 1a between the bodies 1 of a container, and is built in this body 1.

6a is the convection flow control plate prepared in the internal cylinder container 4, and 6b is the convection flow control strip prepared in tubed gap 1a between the internal cylinder container 4 and a body 1. And a raw material is placed below the convection flow control plate 6a in the internal cylinder container 4, while a seed is placed above the plate. To the space volume of the buffer room 5, the real space volume of tubed gap 1a of the body 1 of a container, and the container liner container 4, and the real space volume of the container liner container 4 The concentration of the alkali solution with which was filled up with the alkali solution made into a respectively fixed volume rate, and the buffer room 5 and tubed gap 1a were filled up is set up more thinly than the alkali concentration in the container liner container 4.

In this condition, the body 1 of a container is heated from the outside, and hydrothermal synthesis is performed.

Next, an operation is explained.

Since the buffer room 5 is [a / tubed gap 1] open for free passage with the interior of the container liner container 4 through micropore 4a' again through micropore 5b If the volume in tubed gap 1a and the volume in the container liner container 4 are different a little from a fixed rate or differential pressure is therefore produced within and without the container liner container 4 based on the difference with the solution temperature in tubed gap 1a, and the solution temperature in the container liner container 4 in heating The alkali solution in the buffer room 5 moves into the container liner container 4 or tubed gap 1a, and balance of the inside-and-outside pressure of the

container liner container 4 is maintained.
Moreover, since the alkali solution containing Fe+ ion in tubed gap 1a is mixed with the alkali solution in the buffer room 5 and it enters into the container liner container 4, Fe+ ion is diluted and the effect to the product decreases.
Moreover, since the twist has also made concentration thin at the alkali solution in the container liner container 4, the erosion degree of the body 1 of a container falls, and, as for the alkali solution in tubed gap 1a, generating of Fe+ ion decreases.
Buffer room 5' is shown in Fig. 3 as other examples of structure of a buffer room. Intermediary-ed cube type member 5'a is fixed for micropore 4a' on the inferior surface of tongue of lid 4a of a container liner container, buffer room 5' is formed in it, and micropore 5'b of 0.4mmphi extent is prepared also in the side plate which serves as the upper part from an oil level.
In addition, as for this buffer room 5', it is desirable that inside-and-outside sides are noble metals.
If such buffer room 5' is prepared, the alkali solution containing Fe+ ion which flowed from micropore 4a' is mixed with the alkali solution in buffer room 5', and since Fe+ ion is diluted and it enters into the container liner container 4, it has the almost same operation effectiveness as an example.
Moreover, since a good thing is desired, when heat transfer is alienated, its width of tubed gap 1a is large, heat transfer between the body 1 of a container and the container liner container 4 is made into thermally conductive fitness, and it has permeability and it is filled up in tubed gap 1a, the large ingredient, for example, nickel metal foam, of coefficient of thermal expansion.
Permeability is required for holding the equalization in tubed gap 1a here, and a big coefficient of thermal expansion is required, because adhesion in the wall of the body 1 of a container and the outer wall of the container liner container 4 becomes good with a temperature rise.
(Effect of the invention)
According to this invention, it has the following effectiveness so that I may therefore be understood by the above explanation.
** According to this equipment, there is no generating of Fe+ ion from the container liner container itself which has arranged the seed inside.
** Although a container liner container is comparatively manufactured with thin meat, the stable operation is made, without producing distortion and breakage of a container liner container, since an inside-and-outside pressure can be balanced.
** Since Fe+ ion which the body of a container was eroded by the alkali solution and generated is diluted with the alkali solution of the buffer interior of a room [case / ******] with the high external pressure to a container liner container and enters into a

container liner container, it becomes slight influencing it to a seed of Fe+ ion.

** Since concentration is thin, the alkali solutions with which the body of a container contacts can decrease in number the amount of Fe+ ion which the body of a container is eroded and is generated.

It is ****** so that the product by the above effectiveness and hydrothermal synthesis like good synthetic rock crystal can be supplied.

[Brief Description of the Drawings]

Fig. 1 shows a half-section view of the container for hydrothermal synthesis in the Example, The important section sectional view in which the important section sectional view showing [the sectional view showing the half-section of the example of the container for hydrothermal synthesis which Fig. 1 requires for this invention, and / 2] the lid and buffer room of a container liner container of this example, and Fig. 3 are the same, and showing other examples of structure of the lid of a container liner container and a buffer room, and Fig. 4 shows a sectional view of the conventional container for hydrothermal synthesis.

1: A lid [of the body of a container, a 1a:tubed gap, 4:container liner container, and a 4a:container liner container], 4a':micropore, 4b:bottom plate, 5, and 5':buffer room, 5a, and 5'a:cube type member (wall), 5b, 5'b:micropore, 6a, 6b : convection-current control strip.

Abstract:

PURPOSE: To stably operate an inner cylindrical container by a method wherein a fine hole is provided to the lid of the inner cylindrical container mounted in an externally heated container main body so as to provide a gap and having a noble metal inner surface and a buffer chamber having a fine hole having the lid as one surface is provided to the other wall surface of said lid so as to cover the fine hole of the lid.

CONSTITUTION: A fine hole 4a' is provided to the lid 4a of the inner cylindrical container 4 mounted in a container main body 1 heated from the outside so as to provide a cylindrical gap 1a and having at least an inner surface formed of a noble metal such as silver, gold or platinum and a buffer chamber 5 having a fine hole 5b having the lid as one surface is also provided to the other wall surface of the lid so as to cover the fine hole 4a'. A seed and a raw material are put in the container 4 and the main body 1 is heated from the outside to perform the thermal synthesis of artificial quartz. Since the inner surface of the container 4 is formed of a noble metal in this case, Fe+ ions are generated in the container main body 1 to impart no effect to the seed. Further, the inner cylindrical container generates no strain or damage and can be stably operated.

(19)日本国特許庁（ＪＰ）　(12)特　許　公　報（Ｂ２）　(11)特許出願公告番号

特公平7－22692

(24)(44)公告日　平成７年(1995)３月15日

(51)Int.Cl.[6]		識別記号	庁内整理番号	ＦＩ	技術表示箇所
Ｂ０１Ｊ	3/04		B		
Ｃ３０Ｂ	7/10				
	29/18		8216－4G		

請求項の数３(全 ４ 頁)

(21)出願番号　特願昭63－194338

(22)出願日　昭和63年(1988)８月５日

(65)公開番号　特開平2－43939

(43)公開日　平成２年(1990)２月14日

(71)出願人　999999999
株式会社日本製鋼所
東京都千代田区有楽町１丁目１番２号

(72)発明者　千葉　康太郎
北海道室蘭市茶津町４番地　株式会社日本製鋼所内

(72)発明者　畑中　基秀
北海道室蘭市茶津町４番地　株式会社日本製鋼所内

(72)発明者　河合　政征
北海道室蘭市茶津町４番地　株式会社日本製鋼所内

(74)代理人　弁理士　前田　宏之　(外１名)

審査官　鈴木　紀子

(54)【発明の名称】　水熱合成用容器

1

【特許請求の範囲】

【請求項１】外部から加熱される容器本体内に筒状間隙を設けて内蔵され、少なくとも内面が、銀、金、白金等の貴金属で形成された内筒容器の蓋に微小孔を設け、該微小孔を被う壁を蓋の上面側又は下面側に設けて蓋と壁とで形成される緩衝室となし、該壁面にも微小孔を有せしむることを特徴とする水熱合成用容器。

【請求項２】外部から加熱される容器本体内に筒状間隙を設けて内蔵され、少なくとも内面が銀、金、白金等の貴金属で形成された内筒容器の蓋に微小孔を設け、該微小孔を被う壁を蓋の上面側又は下面側に設けて蓋と壁とで形成される緩衝室となし、該壁面にも微小孔を有せしむる水熱合成用容器に於て、緩衝室の空間容積、前記筒状間隙の実空間容積及び内筒容器の実空間容積に、それぞれ一定の液量割合としてアルカリ溶液を充填し、かつ

2

緩衝室と筒状間隙とに充填したアルカリ溶液の濃度を内筒容器内のアルカリ濃度よりも薄く設定したことを特徴とする水熱合成用容器。

【請求項３】熱伝導性良好な通気性物質を容器本体と内筒容器との間の筒状間隙に充填させた特許請求の範囲第１項又は第２項記載の水熱合成用容器。

【発明の詳細な説明】

（産業上の利用分野）

本発明は、人工水晶の製造等に使用する水熱合成用容器に関する。

（従来の技術）

従来の水熱合成用容器の一例を第４図に示す。21は容器本体、22は蓋、23はクランプ、24は対流制御板、25はヒータであり、各26は熱電対である。容器本体21内には種子Ａと原料Ｂとが収容され、ヒータ25にて加熱された強

アルカリ溶液内に浸漬されている。人工水晶の場合を例にとると、温度350～400℃、圧力1000～1500kg/cm²で使用されるために容器本体21は、高強度、高靭性、かつ耐蝕性に優れた金属材料が使用される。

水熱合成で製造される製品は、電子、光学等の分野への用途が多く、今後益々、小型化、薄帯化される傾向にあるが、製品を粗悪化する主要因として異物の混入がある。特に、容器本体21内面が、強アルカリ溶液によつて侵蝕され、アクマイトなる鉄化合物を生じ、Fe+ イオンが製品へ混入するのが最大の問題点であり、種々の対策がとられている。

一例として種子Ａの表面を隔離する方法があるが、十分ではなく、又保護した面は成長が阻害されて生産効率が低下する。

又、容器本体21の内面を銀、金、白金等で被覆するか、或は上記貴金属で製作した内筒容器を容器本体21に内蔵させて、Fe^{+}イオンの発生自体を阻止することもなされている。

（発明が解決しようとする課題）

容器本体21の内面を、銀、金、白金筒で被覆する場合は、容器本体21の内壁との密着性を確保する上から、水圧拡管又は爆着等の手段を要し、製作が非常に困難である許りでなく、容器本体21の構造によつては、密封個所のシート面の保守に問題を生じ、又銀、金、白金等で製作した内筒容器を容器本体21に内蔵させる場合には、内筒容器に、一方的な外圧が作用しないように、内筒容器の内外の圧力を均等化させる必要があり、このためには、内筒容器の内外の液量割合を同一に保持することが肝要である。しかして、上記両者の適用範囲は、共に実験用の小型容器に限定されていた。

（課題を解決するための手段）

本発明は、上記の事情に鑑みてなされ、大型にして工業用に適した水熱合成用容器を提供するものであり、その構成は次の通りである。すなわち、第１発明は、外部から加熱される容器本体内に筒状間隙を設けて内蔵され、少なくとも内面が、銀、金、白金等の貴金属で形成された内筒容器の蓋に微小孔を設け、該微小孔を被う壁を蓋の上面側又は下面側に設けて蓋と壁とで形成される緩衝室となし、該壁面にも微小孔を有せしむる水熱合成用容器である。

又、第２発明は、外部から加熱される容器本体内に筒状間隙を設けて内蔵され、少なくとも内面が銀、金、白金等の貴金属で形成された内筒容器の蓋に微小孔を設け、該微小孔を被う壁を蓋の上面側又は下面側に設けて蓋と壁とで形成される緩衝室となし、該壁面にも微小孔を有せしむる水熱合成用容器に於て、緩衝室の空間容積、前記筒状間隙の実空間容積及び内筒容器の実空間容積に、それぞれ一定の液量割合としてアルカリ溶液を充填し、かつ緩衝室と筒状間隙とに充填したアルカリ溶液の濃度を内筒容器内のアルカリ濃度よりも薄く設定した水熱合成用容器である。

そして、上記両発明に於て、熱伝導性良好な通気性物質を容器本体と内筒容器との間の筒状間隙に充填させることもある。

（作　用）

次に作用について説明する。

水熱合成用容器の内筒容器の中に種子と原料とを入れて、容器本体を外部から加熱して水熱合成が行われる。その際、内筒容器は、少なくとも内面が貴金属で形成してあるので、容器本体内でFe^{+}イオンが発生して、種子に影響を与えることがない。

又容器本体の筒状間隙と内筒容器内とは、内筒容器の蓋及び緩衝室の壁面に設けた微小孔で連通しているので、内筒容器内外の圧力が均衡し、内筒容器は一方的な外圧を受けない。従つて、内筒容器が歪んだり、破損することがない。

又前記の水熱合成用容器の緩衝室の空間容積、筒状間隙の実空間容積及び内筒容器の実空間容積に、それぞれ一定の液量割合とするアルカリ溶液を充填し、かつ緩衝室及び筒状間隙に充填したアルカリ溶液の濃度を、内筒容器内のアルカリ溶液濃度よりも薄くして使用することによつて、筒状間隙内と内筒容器内との圧力は、実質的に均衡する。しかも、圧力変化が生じると、両微小孔を通して筒状間隙内と内筒容器内の圧力が均衡する。

筒状間隙内の圧力が上昇した場合には、Fe^{+}イオンを含んだ微量のアルカル溶液が微小孔から緩衝室内へ侵入するが、緩衝室内のアルカリ溶液と混合し、Fe^{+}イオンが稀釈されて内筒容器内へ侵入するので、種子に与える影響が微小であり、加えて、筒状間隙に充填したアルカリ溶液の濃度が薄いので容器本体の内面が侵蝕され難く、Fe^{+}イオンの発生量が減少するのと相俟つて種子への影響が更に軽減される。

更に、熱伝導性良好な通気性物質を筒状間隙に充填しておくと、容器本体から内筒容器への熱伝達が良好になされ、かつ筒状間隙内の圧力を均等に保つ。

（実施例）

本発明に係る水熱合成用容器の実施例を図面に基づいて説明する。

第１図に於て、１は外部から加熱される容器本体であり、高強度、高靭性かつ耐蝕性に優れた金属材料からなる。２は容器本体の蓋であり、パツキング2aを介装して、複数個のクランプ３にて容器本体１に着脱可能に装着されている。

４は、有底筒状の内筒容器であり、銀、金、白金等の貴金属製又はTi（チタン）材等の内面に貴金属を合せ材とした材料からなつている。着脱可能な内筒容器の蓋4a及び底板4bも同様の材料からなり、該蓋4aのほぼ中央部には、第２図に詳示するように微小孔4a′を設けてある。この微小孔4a′は、圧力が作用しない状態では、表面張力によつて自重では液体が流下しない大きさであり、具

体的には0.4mmφ程度である。そして、該容器の蓋4aの上面に、微小孔4a′を被つて箱形部材5aを固着して緩衝室５を形成し、この箱型部材5aの天板にも0.4mmφ程度の微小孔5bを設けてある。なお、この微小孔5bの位置は、緩衝室５に充填される液体の液面よりも高い位置とする。

又、緩衝室５の少なくとも内面は貴金属で形成されている。

上記の構成からなる内筒容器４が容器本体１との間に筒状間隙1aを設けて該本体１に内蔵されている。

6aは内筒容器４内に設けられた対流制御板であり、6bは内筒容器４と本体１との間の筒状間隙1aに設けられた対流制御板である。そして、内筒容器４内の対流制御板6aの上方に種子を、下方に原料をそれぞれ収容し、緩衝室５の空間容積及び容器本体１と内筒容器４との筒状間隙1aの実空間容積及び内筒容器４の実空間容積に、それぞれ一定の液量割合とするアルカリ溶液を充填し、かつ緩衝室５及び筒状間隙1aに充填したアルカリ溶液の濃度を、内筒容器４内のアルカリ濃度よりも薄く設定する。この状態にて、容器本体１を外部から加熱して、水熱合成を行う。

次に作用を説明する。

緩衝室５は、微小孔5bを介して筒状間隙1aと又微小孔4a′を介して内筒容器４の内部と連通しているので、筒状間隙1a内の液量と内筒容器４内の液量とが一定割合から若干相違し、或は加熱によつて筒状間隙1a内の液温と内筒容器４内の液温との相違に基づいて、内筒容器４の内外に圧力差を生ずると、緩衝室５内のアルカリ溶液が、内筒容器４内又は筒状間隙1a内へと移動して、内筒容器４の内外圧力の均衡が保たれる。

又筒状間隙1a内のFe^{+}イオンを含んだアルカリ溶液は、緩衝室５内のアルカリ溶液と混合して内筒容器４内へ入るので、Fe^{+}イオンは稀釈されており、その製品に対する影響が減少する。

又、筒状間隙1a内のアルカリ溶液は、内筒容器４内のアルカリ溶液によりも濃度を薄くしてあるので、容器本体１の侵蝕度合が低下し、Fe^{+}イオンの発生が減少する。

第３図に、緩衝室の他の構造例として緩衝室５′を示す。内筒容器の蓋4aの下面に、微小孔4a′を被つて箱形部材５′ａを固着して緩衝室５′を形成し、液面よりも上方となる側板にも0.4mmφ程度の微小孔５′ｂを設けてある。

なお、この緩衝室５′は、内外面共に貴金属であることが好ましい。

このような緩衝室５′を設ければ、微小孔4a′から流入したFe^{+}イオンを含むアルカリ溶液は、緩衝室５′内のアルカリ溶液と混合して、Fe^{+}イオンが希釈されて内筒容器４内へ入るので実施例とほぼ同様な作用効果を有す。

又、容器本体１と内筒容器４との間の熱伝達は、良好であることが望まれるので、筒状間隙1aの巾が大きく、熱伝達が疎外されるような場合には、熱伝導性良好にして通気性を有し、かつ熱膨張率の大きい材料、例えばNi金属発泡体を筒状間隙1a内に充填する。

ここで、通気性を要求するのは、筒状間隙1a内の均圧を保持するためであり、又大きな熱膨張率を要求するのは、温度上昇と共に容器本体１の内壁及び内筒容器４の外壁への密着が良好となるためである。

（発明の効果）

以上の説明によつて理解されるように、本発明によれば、下記の効果を有する。

①本装置によれば、種子を内部に配置した内筒容器自体からのFe^{+}イオンの発生がない。

②内筒容器は、比較的薄肉で製作されるが、内外圧力を均衡できるので、内筒容器の歪や破損を生じることなく、安定した操業がなされる。

③容器本体がアルカリ溶液に侵蝕されて発生したFe^{+}イオンは、内筒容器に対する外圧が高くなつた場合には、緩衝室内のアルカリ溶液で稀釈されて内筒容器内へ入るので、種子に対するFe^{+}イオンの影響が軽微となる。

④容器本体が接触するアルカリ溶液は、濃度が薄いので容器本体が侵蝕されて発生するFe^{+}イオンの量を減少できる。

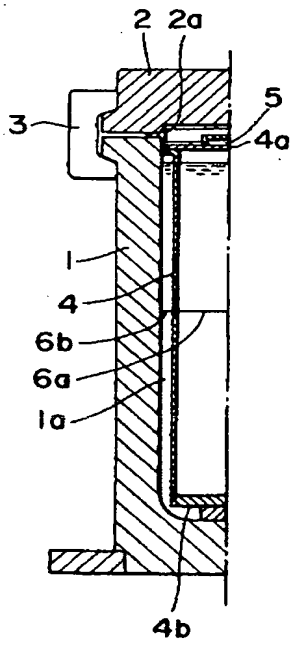
以上の効果、良質な人工水晶のような水熱合成による製品を供給できるようになつた。

【図面の簡単な説明】

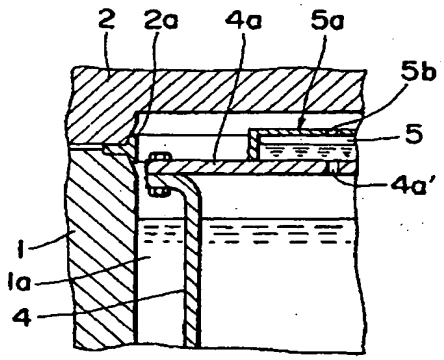
第１図は、本発明に係る水熱合成用容器の実施例の半部を示す断面図、第２図は、本実施例の内筒容器の蓋と緩衝室とを示す要部断面図、第３図は、同じく内筒容器の蓋と緩衝室との他の構造例を示す要部断面図、第４図は、従来の水熱合成用容器の断面図である。

1:容器本体、1a:筒状間隙、4:内筒容器、4a:内筒容器の蓋、4a′：微小孔、4b:底板、5,5′：緩衝室、5a,5′a:箱形部材（壁）、5b,5′b:微小孔、6a,6b:対流制御板。

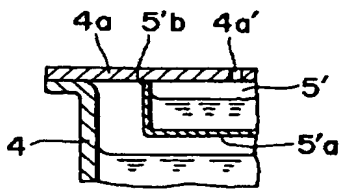
【第1図】



【第2図】



【第3図】



【第4図】